

**We Claim:**

1. Crystalline tiotropium bromide monohydrate.
2. Crystalline tiotropium bromide monohydrate according to claim 1, having an endothermic peak at  $230^{\circ}\text{C} \pm 5^{\circ}\text{C}$  occurring during thermal analysis using DSC at a heating rate of 10 K/min.
3. Crystalline tiotropium bromide monohydrate according to claim 1, having an IR spectrum comprising bands at wave numbers 3570, 3410, 3105, 1730, 1260, 1035, and  $720\text{ cm}^{-1}$ .
4. Crystalline tiotropium bromide monohydrate according to claim 2, having an IR spectrum comprising bands at wave numbers 3570, 3410, 3105, 1730, 1260, 1035, and  $720\text{ cm}^{-1}$ .
5. Crystalline tiotropium bromide monohydrate according to claim 1, having a single monoclinic cell having the following dimensions:  $a = 18.0774\text{ \AA}$ ,  $b = 11.9711\text{ \AA}$ ,  $c = 9.9321\text{ \AA}$ ,  $\beta = 102.691^{\circ}$ , and  $V = 2096.96\text{ \AA}^3$ .
6. Crystalline tiotropium bromide monohydrate according to claim 2, having a single monoclinic cell having the following dimensions:  $a = 18.0774\text{ \AA}$ ,  $b = 11.9711\text{ \AA}$ ,  $c = 9.9321\text{ \AA}$ ,  $\beta = 102.691^{\circ}$ , and  $V = 2096.96\text{ \AA}^3$ .
7. Crystalline tiotropium bromide monohydrate according to claim 3, having a single monoclinic cell having the following dimensions:  $a = 18.0774\text{ \AA}$ ,  $b = 11.9711\text{ \AA}$ ,  $c = 9.9321\text{ \AA}$ ,  $\beta = 102.691^{\circ}$ , and  $V = 2096.96\text{ \AA}^3$ .
8. Crystalline tiotropium bromide monohydrate according to claim 4, having a single monoclinic cell having the following dimensions:  $a = 18.0774\text{ \AA}$ ,  $b = 11.9711\text{ \AA}$ ,  $c = 9.9321\text{ \AA}$ ,  $\beta = 102.691^{\circ}$ , and  $V = 2096.96\text{ \AA}^3$ .

9. A process for preparing crystalline tiotropium bromide monohydrate, the process comprising:

- (a) dissolving tiotropium bromide in water to obtain a solution;
- (b) heating the resulting solution;
- (c) adding activated charcoal to the heated solution;
- (d) removing the activated charcoal; and
- (e) allowing the solution to slowly cool to obtain crystalline tiotropium bromide monohydrate.

10. A process for preparing crystalline tiotropium bromide monohydrate, the process comprising:

- (a) dissolving tiotropium bromide in water to obtain a solution;
- (b) heating the resulting solution to more than 50°C;
- (c) adding activated charcoal to the heated solution;
- (d) removing the activated charcoal; and
- (e) allowing the solution to slowly cool to obtain crystalline tiotropium bromide monohydrate.

11. The process according to claim 10, wherein 0.4 to 1.5 kg of water are used per mole of tiotropium bromide in step (a).

12. The process according to claim 11, wherein 10 g to 50 g of activated charcoal per mole of tiotropium bromide is added in step (c).

13. The process according to claim 12, wherein the activated charcoal added in step (c) is stirred for between 5 and 60 minutes before it is removed in step (d).

14. The process according to claim 13, wherein step (d) is performed by filtration of the solution.

15. The process according to claim 14, wherein the solution of step (e) is cooled to a temperature of 20°C-25°C at a cooling rate of 1 to 10°C per 10 to 30 minutes.
16. A pharmaceutical composition comprising an effective amount of crystalline tiotropium bromide monohydrate according claim 1 and a pharmaceutically acceptable excipient.
17. A method for treatment of diseases in which the administration of an anticholinergic agent may have a therapeutic benefit, in a patient in need of such treatment, which method comprises administering the patient an effective amount of a compound according to claim 1.
18. The method according to claim 17, wherein the disease is asthma or COPD.
19. A process for preparing crystalline hydrates of tiotropium bromide, the process comprising:
- (a) dissolving tiotropium bromide in water to obtain a solution;
  - (b) heating the resulting solution; and
  - (c) allowing the solution to slowly cool to obtain crystalline hydrates of tiotropium bromide.
20. A process for preparing crystalline hydrates of tiotropium bromide, the process comprising:
- (a) dissolving tiotropium bromide in water to obtain a solution;
  - (b) heating the solution of step (a);
  - (c) adding activated charcoal to the heated solution of step (b);
  - (d) removing the activated charcoal from the solution of step (c); and
  - (e) allowing the solution to slowly cool to obtain crystalline hydrates of tiotropium bromide.
21. The process of claim 20, wherein the solution of step (a) is heated to more than 50°C.